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Re: N00173-98-1-G014

Dear Dr. Soulen:

Pursuant to applicable grant requirements I enclose one (1) original and two (2) copies of the annual performance report for the referenced award. This report covers the period July 1, 1998 to June 30, 1999.

Sincerely,

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Sr. Projects Officer

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13. ABSTRACT *(Maximum 200 words)*

During the period covered by this report (July 1998 – June 1999), we initiated the application of crystal ion slicing to single-crystal potassium tantalate and strontium titanate. Transport-of-ions-in-matter (TRIM) simulations and ion implantation at 3.8MeV and various dosages were done and removal of the sacrificial layer tested in a number of etchants. Potassium tantalate films have been fabricated and characterized. Sacrificial-layer etching studies in $SrTiO_3$ for (110) crystals were also carried out.

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Epitaxial Liftoff for Fully Single-Crystal Ferroelectric Thin Films

Progress Report (7/1/98-6/30/99) for DARPA/FAME project - Contract N00173-98-1-G014

During the period covered by this report (July 1998 – June 1999), we demonstrated the first crystal ion slicing of single-crystal potassium tantalate and strontium titanate; this constitutes the first fully single crystals thin films made of these materials. Transport-of-ions-in-matter (TRIM) simulations and ion implantation at 3.8MeV and various dosages were done and removal of the sacrificial layer tested in a number of etchants. Potassium tantalate films have been fabricated and characterized and sacrificial-layer etching studies in $SrTiO_3$ for (110) crystals were also carried out. In addition, a new approach to enhancing ion slicing has been developed using $LiNbO_3$ samples as model crystals.

Potassium Tantalate

Single-Crystal Film Fabrication

Etch Selectivity and Implantation Dosage

10μm-thick films were fabricated at an implantation energy of 3.8MeV and a dosage of 1×10^{16} cm^{-2}. Other dosage levels were also explored. These experiments showed that exfoliation occurred above the sacrificial layer for levels over 1×10^{16} cm^{-2}. Below this point, the etch selectivity in diluted HCl increased dramatically between 5×10^{15} cm^{-2} and 1×10^{16} cm^{-2}. The undercut evolution rate ranged from less than a micron per hour at the low end, to 300 μm/h near the exfoliation threshold. At high dosages the films tended to cleave during etching, possibly as a result of implantation-induced stress. Films up to 0.5×1.0 mm^2 in size have been fabricated so far. The effect of heat treatment on etch selectivity is yet to be investigated.

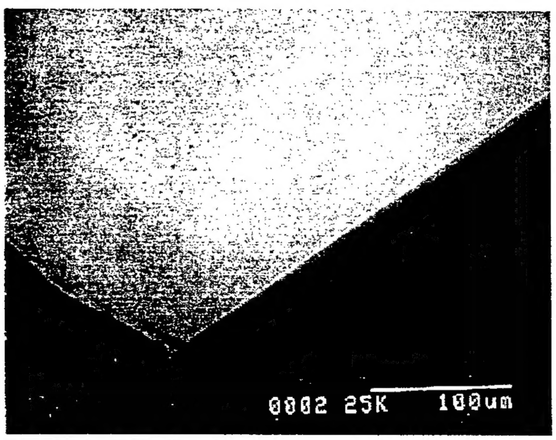


Fig.1(a). $KTaO_3$ single-crystal film.

Figure 1(a) shows a fully detached $KTaO_3$ film. While the film's top surface is smooth, its undersurface exhibits regular narrow grooves, less than 0.2 μm in depth. A sample film's underside is displayed in Fig. 1(b).

Crystallographic Studies

We have initiated a series of crystallographic tests to probe the effect of implantation dosage on crystal structure in $KTaO_3$. Our aim is to better understand the evolution of strain leading to exfoliation at a threshold dosage of 1×10^{16} cm^{-2}. X-ray rocking curve measurements have been obtained at three different dosages below threshold. These experiments were done in collaboration with A.Kumar and H. Bakhru at SUNY Albany. The plots show a pronounced shift in peak position in the implanted region, from $2\theta = 22.5°$ for the unimplanted material, to $2\theta = 22.4°$ at 5×10^{15} cm^{-2} dosage. This corresponds to a strain of $\Delta a = 0.018$A in the lattice parameter a at the implantation layer. Rutherford back scattering (RBS) measurements are also planned in these samples.

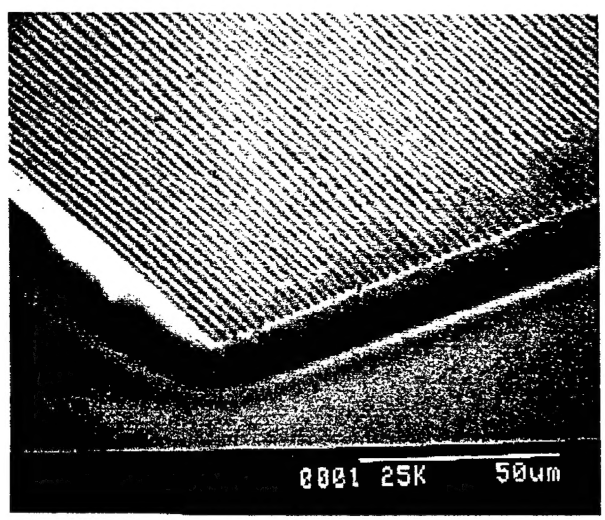


Fig.1(b). Underside of $KTaO_3$ film.

Capacitance of Free-Standing $KTaO_3$ Films

It is important to study the dielectric properties of single-crystal thin ferroelectric films, to determine whether they perform as bulk material, or whether deviations are brought about by purely geometrical factors. This is an issue central to the goals of the FAME program. In particular, are these films highly tunable, and are the values of dielectric constant and loss tangent comparable to that of bulk material? To answer these questions, we have started a study of the electrical properties in crystal ion sliced $KTaO_3$ films in collaboration with R. Guo, A. Bhalla and L.E. Cross of Penn State University. Capacitance measurements have been done initially on films with electrodes placed on top and bottom surfaces using conductive epoxy. Low-temperature data was obtained showing the expected Curie-Weiss behavior, with a Curie point near 20K as in single-crystal bulk material. A secondary peak, observable near 60K in Fig. 2 has been tentatively identified with the presence of strain leading to weak ferroelectric behavior. Further tests are underway, including prior annealing to eliminate residual implantation strain. Presently we are preparing the deposition of aluminum electrodes to improve electrical contact. This is important to get an accurate reading of the absolute value in the dielectric constant. Figure 1(b) shows the film placed on a 1μm-thick photoresist layer to prevent shorting during deposition.

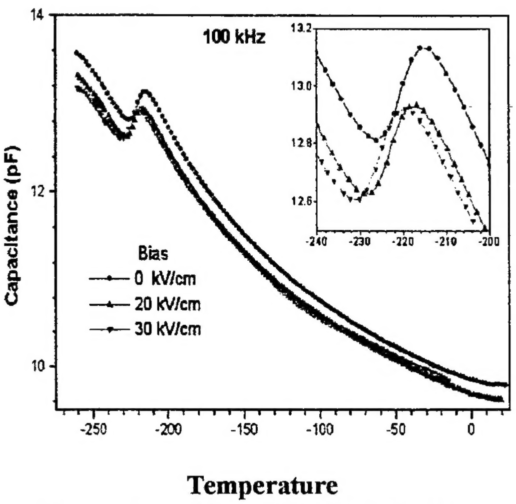


Fig. 2. Capacitance in a $KTaO_3$ film

Strontium Titanate

Etch Selectivity and Crystal Orientation

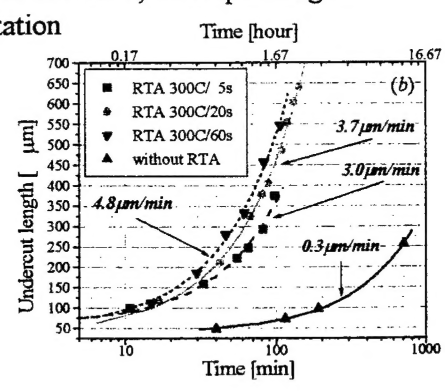
(110) crystals were implanted with He^+ at 3.8MeV, 5×10^{16} cm^{-2} and 1×10^{17} cm^{-2}. An undercut etch rate of 33 μm/h was observed at the lower dosage and 65 μm/h for 1×10^{17} cm^{-2} along the (1$\underline{1}$0) direction.

A lower etch rate was measured for (001), indicating crystallographic dependence to the etch selectivity. Some implantation induced cleavage occurred during the etching. Annealing studies are planned.

Etch Selectivity Enhancement

$LiNbO_3$ as a Model Ferroelectric

Rapid thermal treatment has been found to reduce the time needed for film detachment in ferroelectric fields by a factor as large as 100. This makes it possible to fabricate large films (0.5 x 1cm^2) of excellent quality in just a matter of a few hours. Using $LiNbO_3$ as a model material, two different crystallographic orientations were studied: 1) c-cut and 2) phase-matching-cut for second harmonic generation at 1.55μm. Several ionic energies ranging from 3.18 MeV to 3.8 MeV, corresponding to penetration depths 8 to 10μm, were used to probe the effect of implantation energy on etch selectivity. Both crystal orientations exhibited significant selectivity enhancement, confirming that heat treatment speeds up film fabrication regardless of sample cut. Secondary ion mass spectroscopy (SIMS) showed that the He^+ distribution of implanted ions evolved toward sharper and narrower spectra. Heat treatment thus yields an increase in internal pressure at the sacrificial layer.



Talks

1. MRS '99, Materials Research Society Symposium, "Helium-Implantation Induced Layer Detachment and Electrical Properties of Single-Crystal Potassium Tantalate Films," M. Levy, R.M. Osgood, Jr., R. Guo, A. Bhalla, L.E.Cross, A. Kumar, and H. Bakhru, Boston, MA, Nov 29- Dec 3, 1999.

2. MRS '99, Materials Research Society Symposium, "Narrow-Linewidth Yttrium Iron Garnet Films for Heterogeneous Integration," M. Levy, R.M. Osgood, Jr., F. J. Rachford, A. Kumar, and H. Bakhru, Boston, MA, Nov 29-Dec 3, 1999.

3. CLEO/QELS'99 Meeting, "Liftoff and Rapid Processing of 10mm-Thick Single-Crystal $LiNbO_3$ Films," A.M. Radojevic, M. Levy, and R.M. Osgood, Jr., Baltimore, MD, May 23-28, 1999.

4. City College of NY, Physics Department Colloquium, "Crystal Ion Slicing for Integrated Photonics," Miguel Levy, New York, NY, September 9, 1998.

5. Michigan Technological University, (invited talk), "Crystal Ion Slicing for Integrated Circuit Applications," Miguel Levy, Houghton, MI, September 10, 1998.

Papers

1. A.M. Radojevic, M. Levy, R. M. Osgood Jr., K. Atul, H. Bakhru, C. Tian and C. Evans, "Large Etch-Selectivity Enhancement in the Epitaxial Liftoff of Single-Crystal $LiNbO_3$ Films," Appl. Phys. Lett. 74, 3197 (1999).

2. M. Levy, R.M. Osgood Jr., R. Liu, L.E. Cross, G. S. Cargill III, A. Kumar, and H. Bakhru, "Fabrication of Single-Crystal Lithium Niobate Films by Crystal Ion Slicing," Appl. Phys. Lett. 73, 2293 (1998).

3. R. Liu, R. Guo, A. S. Bhalla, L.E. Cross, M. Levy, R.M. Osgood Jr., A. Kumar, and H. Bakhru, "Dielectric and Pyroelectric Properties of Crystal Ion Sliced (CIS) $LiNbO_3$ Thin Films," accepted for publication in Ferroelectrics.

4. R. Liu, R. Guo, A.S. Bhalla, L.E. Cross, M. Levy, and R.M. Osgood, Jr., "Optical Observation of Dynamic Ferroelectric Phase Transition and Static Domain Structures in Crystal Ion Siced (CIS) $LiNbO_3$ Film," Materials Letters 39, 264-267 (1999).